

I CLAIM:

1. A method of speciated isotope dilution measurement of a sample comprising

providing at least one predetermined stable isotope,

preparing an isotopic spike by converting said stable isotope to a speciated enriched isotope corresponding to the species to be measured in said sample,

spiking the sample containing said specie to be measured,

equilibrating said isotopic spiked species with said species to be measured,

separating at least a portion of said species from said sample,

making isotope ratio determinations for each said specie to be measured and mathematically deconvoluting said species concentration while correcting for species conversion, and

effecting said mathematical deconvolution while correcting for incomplete separation of said species from said sample.

2. The method of speciated isotope dilution measurement of claim 1 including

employing said method on more than one said species to be measured simultaneously.

3. The method of speciated isotope dilution measurement of claim 2 including

employing said method in quantifying Cr(III) and Cr(VI).

4. The method of speciated isotope dilution measurement of claim 2 including

effecting said mathematical deconvolution employing the following formulas:

$$R_{50/52}^{III} = \frac{(^{50}A_x C_x^{III} W_x + ^{50}A_s C_s^{III} W_s^{III})(1-\alpha) + (^{50}A_x C_x^{VI} W_x + ^{50}A_s C_s^{VI} W_s^{VI})\beta}{(^{52}A_x C_x^{III} W_x + ^{52}A_s C_s^{III} W_s^{III})(1-\alpha) + (^{52}A_x C_x^{VI} W_x + ^{52}A_s C_s^{VI} W_s^{VI})\beta}$$

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$$R_{53/52}^{III} = \frac{(^{53}A_x C_x^{III} W_x + ^{53}A_s C_s^{III} W_s^{III})(1-\alpha) + (^{53}A_x C_x^{VI} W_x + ^{53}A_s C_s^{VI} W_s^{VI})\beta}{(^{52}A_x C_x^{III} W_x + ^{52}A_s C_s^{III} W_s^{III})(1-\alpha) + (^{52}A_x C_x^{VI} W_x + ^{52}A_s C_s^{VI} W_s^{VI})\beta}$$

$$R_{50/52}^{VI} = \frac{(^{50}A_x C_x^{III} W_x + ^{50}A_s C_s^{III} W_s^{III})\alpha + (^{50}A_x C_x^{VI} W_x + ^{50}A_s C_s^{VI} W_s^{VI})(1-\beta)}{(^{52}A_x C_x^{III} W_x + ^{52}A_s C_s^{III} W_s^{III})\alpha + (^{52}A_x C_x^{VI} W_x + ^{52}A_s C_s^{VI} W_s^{VI})(1-\beta)}$$

$$R_{53/52}^{VI} = \frac{(^{53}A_x C_x^{III} W_x + ^{53}A_s C_s^{III} W_s^{III})\alpha + (^{53}A_x C_x^{VI} W_x + ^{53}A_s C_s^{VI} W_s^{VI})(1-\beta)}{(^{52}A_x C_x^{III} W_x + ^{52}A_s C_s^{III} W_s^{III})\alpha + (^{52}A_x C_x^{VI} W_x + ^{52}A_s C_s^{VI} W_s^{VI})(1-\beta)}$$

where,

$R_{50/52}^{III}$ is the measured isotope ratio of ^{50}Cr to ^{52}Cr of Cr(III) in the spiked sample

$^{50}A_x$ is the natural atomic fraction of ^{50}Cr in the sample

C_x^{III} is the concentration of Cr(III) in the sample ($\mu\text{mole/g}$, unknown)

W_x is the weight of the sample in grams

$^{50}A_s^{III}$ is the atomic fraction of ^{50}Cr in the isotopic spike: $^{50}\text{Cr(III)}$

C_s^{III} is the concentration of Cr(III) in the $^{50}\text{Cr(III)}$ spike ($\mu\text{mole/g}$)

W_s^{III} is the weight of the $^{50}\text{Cr(III)}$ spike in grams

C_x^{VI} is the concentration of Cr(VI) in the sample ($\mu\text{mole/g}$, unknown)

α is the percentage of Cr(III) oxidized to Cr(VI) after spiking (unknown)

β is the percentage of Cr(VI) reduced to Cr(III) after spiking (unknown)

5. The method of speciated isotope dilution measurement of claim 1 including

employing a mass spectrometer to determine said isotopic element ratios.

6. The method of speciated isotope dilution measurement of claim 1 including

tagging said enriched isotope with an isotopic tag in the same speciated form as the species to be measured.

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7. The method of speciated isotope dilution measurement of claim
1 including
employing time resolution chromatography to effect said
separation.
8. The method of speciated isotope dilution measurement of claim
1 including
employing said process on a sample which has experienced
specie conversion prior to separation.
9. The method of speciated isotope dilution measurement of claim
1 including
effecting said equilibrium in an aqueous solution.
10. The method of speciated isotope dilution measurement of claim
1 including
employing said process on an incompletely separated specie.
11. The method of speciated isotope dilution measurement of claim
3 including
effecting said separation after reduction of a substantial portion
of Cr(VI) to Cr(III).
12. The method of speciated isotope dilution measurement of claim
1 including
employing said process on a soil sample.
13. The method of speciated isotope dilution measurement of claim
1 including
employing said process on an aqueous sample.
14. The method of speciated isotope dilution measurement of claim
1 including
employing said process on solid waste from a chromite ore
processing system.
15. The method of speciated isotope dilution measurement of claim
1 including
storing said sample after said equilibrating step and prior to said
separating step.

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employing said method to validate said tests which are not independently capable of compensating for incomplete species extraction or species conversion.

32. The method of speciated isotope dilution measurement of claim 1 including

performing said method on a species which due to incomplete separation, loss, conversion or degradation has less than 100 percent of said species separated.

33. The method of speciated isotope dilution measurement of claim 32 including

effecting said separation of at least about 5 to 10 percent of said species.

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